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(56) Documents Cited

EP 0658361 A

Abstracts of JP 081922001 A (Sanpuku Co.)

(58) Field of Search

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(54) Dilution stable antifoam emulsion concentrate

(57) A water-in-oil emulsion comprising a silicone oil (preferably a polydimethylsiloxane), silica, emulsifiers, and thickening agents having the property that upon dilution the emulsion inverts to an oil-in-Water emulsion useful as an antifoam agent.

The emulsifier has an HLB of between 2 and 20 and is preferably an ester of oleic acid, especially an ester derived from glycerin.

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DILUTION STABLE ANTIFOAM EMULSION CONCENTRATES

The present invention relates to an antifoam concentrate that is a water-in-oil emulsion when concentrated but when diluted inverts and forms an oil-in-water emulsion that continues to function as an antifoam.

Background of the Invention

Antifoam compositions are materials that when added to a foaming liquid equilibrate or accelerate the rate of foam collapse relative to the rate of foam formation. Usually such materials are added in small concentrations. Antifoaming agents have found application in a wide variety of manufacturing and treating processes, e.g. paints and latexes, coating processes, textiles, fermentation processes, polymer manufacture, cleaning compounds, pulp and paper, waste water treatment, and cooling towers.

It is generally a desired characteristic that antifoam agents be inert to the product or system in which it is used, i.e. that it have no adverse effect. The components of an antifoam composition generally consist of primary and secondary antifoam agents, a carrier, an emulsifier, and if desired a stabilizing agent. The primary antifoam agent is usually a hydrophobic silica dispersed in an oil such as a hydrocarbon wax, a fatty acid, fatty acid ester, or fatty acid amide. It is the combination of the two ingredients that produces the antifoam action because hydrophobic silica by itself is inactive for collapsing foams. The secondary antifoam is usually a silicone or fatty alcohol or ester which increases the antifoam effect of the primary antifoam

agent. Carriers that are typically employed include hydrocarbon oils and solvents, water, and fatty alcohols or esters. Emulsifiers function to introduce the primary and secondary antifoam agents into the system being treated.

There are two basic types of antifoam compositions that comprise
5 silicone compounds. The first type is a composition that utilizes materials that have a particular lower critical solubility temperature such as silicone polyethers. The mode of use of such materials involves using the composition at a temperature above its lower critical solubility temperature which is a temperature range where the material loses its solubility in water
10 and therefore functions as an antifoaming agent. Because of the restriction on the lower critical solubility temperature, these types of antifoam compositions possess only limited utility because they function only within a narrow temperature range.

U. S. patent 5,106,535 teaches a silicone based defoaming composition
15 that comprises a polyoxyalkylene substituted organopolysiloxane, a dimethyl silicone fluid and a finely divided silica powder. In order to function as a defoaming agent the composition of the '535 patent requires a surface active agent for dispersal in aqueous systems to control foaming. The requirement that a surface active agent is necessary for dispersal of the defoaming
20 composition has been modified by the incorporation of a second polyoxyalkylene modified silicone into the composition as taught in U. S. patent 5,244,599 which acts as a surface active agent.

U. S. patent 5,169,561 describes an unusual antifoam concentrate where the finely divided silica has been chemically treated with an anti-microbial
25 agent to render the composition resistant to bacterial spoilage.

The second and more traditional type of antifoam composition is an oil-in-water emulsion prepared by emulsifying a silicone polymer and finely divided silica. While these types of emulsions are generally very effective as antifoam agents they are not stable to dilution. Because such oil-in-water

emulsions that function as antifoaming agents are thermodynamically unstable, thickening agents to increase the viscosity of the aqueous phase are added to retard the particle settling rate which increases the shelf stability of the emulsion. When these emulsions are diluted with water, the viscosity is reduced by the dilution and the emulsion becomes unstable.

There is thus a need for a traditional antifoaming agent that can be diluted into a wide range of concentrations and remain effective as an antifoaming agent.

Summary of the Invention

We now disclose a water-in-oil emulsion that functions as an antifoam agent and, when diluted, inverts to an oil-in-water emulsion that continues to function as an antifoaming agent through a wide range of concentrations.

Thus the present invention provides for a water-in-oil emulsion comprising:

(a) from about 1 to about 99 percent by weight of a polyorganosiloxane of the general formula



where $M = R^1R^2R^3SiO_{1/2}$ where R^1 , R^2 , and R^3 are each independently selected monovalent one to forty carbon atom hydrocarbon radicals

where $D = R^4R^5SiO_{2/2}$ where R^4 and R^5 are each independently selected monovalent one to forty carbon atom hydrocarbon radicals, with the stoichiometric subscript, x , ranging from about 1 to about 10,000 wherein the viscosity of the polyorganosiloxane ranges from about 1 to about 10,000,000 centipoise at 25 °C;

(b) from about 0.01 to about 50.0 percent by weight of a finely divided silica having a size ranging from about 0.001 to about 1,000 microns average particle diameter;

(c) from about 0.10 to about 50.0 percent by weight of an emulsifier or mixture of emulsifiers having on average a hydrophilic lipophilic balance ratio ranging from about 2 to about 20; and

5 (d) from about 0.001 to about 20.0 percent by weight of a thickening agent or mixture of a water soluble thickening agent selected from the group consisting of polyacrylates, polyamides, polyamines, styrene sulfonate polymers, polyethylene oxides and cellulose derivatives having a molecular weight ranging from about 100 to about 100,000,00 dalton; and

10 (e) from slightly greater than 0 to about 20.0 percent by weight of water; wherein when said water-in-oil emulsion is added to a quantity of water greater in weight than the weight of said water-in-oil emulsion said emulsion inverts to an oil-in-water emulsion wherein said oil-in-water emulsion reduces foaming.

The present invention also provides for a method of reducing foaming
15 comprising:

(a) preparing a water-in-oil emulsion comprising:

(i) from about 1 to about 99 percent by weight of a polyorganosiloxane of the general formula



20 where $M = R^1R^2R^3SiO_{1/2}$ where R^1 , R^2 , and R^3 are each independently selected monovalent one to forty carbon atom hydrocarbon radicals

25 where $D = R^4R^5SiO_{2/2}$ where R^4 and R^5 are each independently selected monovalent one to forty carbon atom hydrocarbon radicals, with the stoichiometric subscript, x , ranging from about 1 to about 10,000 wherein the viscosity of the polyorganosiloxane ranges from about 1 to about 10,000,000 centipoise at 25 °C;

(ii) from about 0.01 to about 50.0 percent by weight of a finely divided silica having a size ranging from about 0.001 to about 1,000 microns average particle diameter;

(iii) from about 0.10 to about 50.0 percent by weight of an emulsifier or mixture of emulsifiers having on average a hydrophilic lipophilic balance ratio ranging from about 2 to about 20;

(iv) from about 0.001 to about 20.0 percent by weight of a thickening agent or mixture of a water soluble thickening agent selected from the group consisting of polyacrylates, polyamides, polyamines, styrene sulfonate polymers, polyethylene oxides and cellulose derivatives having a molecular weight ranging from about 100 to about 100,000,00 dalton; and

(v) from slightly greater than 0 to about 20.0 percent by weight of water; wherein when said water-in-oil emulsion is added to a quantity of water greater in weight than the weight of said water-in-oil emulsion said emulsion inverts to an oil-in-water emulsion wherein said oil-in-water emulsion reduces foaming;

(b) adding the water-in-oil emulsion to a quantity of water having a weight greater than the quantity of the water-in-oil emulsion; and

(c) inverting the emulsion from a water-in-oil emulsion to an oil-in-water emulsion whereby the rate of foam collapse exceeds the rate of foam formation.

Detailed Description of the Invention

The present invention is a water-in-oil emulsion comprising:

(a) from about 1 to about 99 percent by weight, preferably from about 5 to about 95 percent by weight, more preferably from about 25 to about 90 percent by weight, and most preferably from about 60 to about 85 percent by weight of a polyorganosiloxane of the general formula

MD_xM

where M = R¹R²R³SiO_{1/2} where R¹, R², and R³ are each independently monovalent one to forty carbon atom hydrocarbon radicals, preferably one to twenty carbon atom hydrocarbon radicals, more preferably one to ten carbon atom radicals, and most preferably methyl, ethyl, n-propyl, i-propyl, tri-
5 fluoro-propyl, n-butyl, i-butyl, sec-butyl, n-pentyl, i-pentyl, neo-pentyl, n-hexyl, i-hexyl, phenyl, and benzyl;

where D = R⁴R⁵SiO₂/2 where R⁴ and R⁵ are each independently monovalent one to forty carbon atom hydrocarbon radicals, preferably one to
10 twenty carbon atom hydrocarbon radicals, more preferably one to ten carbon atom radicals, and most preferably methyl, ethyl, n-propyl, i-propyl, tri-fluoro-propyl, n-butyl, i-butyl, sec-butyl, n-pentyl, i-pentyl, neo-pentyl, n-hexyl, i-hexyl, phenyl, and benzyl with the stoichiometric subscript, x, ranging from about 1 to about 10,000, preferably from about 10 to about
15 5,000, more preferably from about 50 to about 3,000, and most preferably from about 100 to about 1,000, wherein the viscosity of the polyorganosiloxane ranges from about 1 to about 10,000,000 centipoise at 25°C, preferably from about 10 to about 1,000,000 centipoise at 25 °C, more preferably from about 50 to about 100,000 centipoise at 25 °C, and most
20 preferably from about 100 to about 10,000 centipoise at 25 °C;

(b) from about 0.01 to about 50.0 percent by weight, preferably from about 0.10 to about 30.0 percent by weight, more preferably from about 0.50 to about 20.0 percent by weight, and most preferably from about 1.0 to about 10.0 percent by weight of a finely divided silica having a size ranging from
25 about 0.001 to about 1,000 microns average particle diameter, preferably from about 0.010 to about 100 microns average particle diameter, more preferably from about 0.050 to about 50 microns average particle diameter, and most preferably from about 0.100 to about 10 microns average particle diameter;

(c) from about 0.10 to about 50.0 percent by weight, preferably from about 1.0 to about 40.0 percent by weight, more preferably from about 5.0 to about 30.0 percent by weight, and most preferably from about 10.0 to about 20.0 percent by weight of an emulsifier or mixture of emulsifiers having on average a hydrophilic lipophilic balance (HLB) ratio ranging from about 2 to about 20, preferably from about 3 to about 18, more preferably from about 4 to about 16, and most preferably from about 5 to about 14; and

(d) from about 0.001 to about 20.0 percent by weight, preferably from about 0.010 to about 15.0 percent by weight, more preferably from about 0.050 to about 10.0 percent by weight, and most preferably from about 0.10 to about 5.0 percent by weight of a thickening agent or mixture of thickening agents that are water soluble polymers selected from the group consisting of polyacrylates, polyamides, polyamines, styrene sulfonate polymers, polyethylene oxides and cellulose derivatives having a molecular weight ranging from about 100 to about 100,000,00 dalton; preferably from about 1,000 to about 50,000,000 dalton; more preferably from about 10,000 to about 10,000,000 dalton; and most preferably from about 100,000 to about 5,000,000 dalton; and

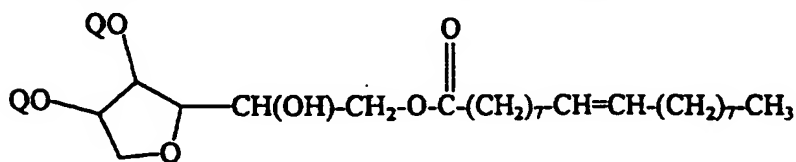
(e) from slightly greater than 0 to about 20.0 percent by weight, preferably from about 0.01 to about 15.0 percent by weight, more preferably from about 0.050 to about 10.0 percent by weight, and most preferably from about 0.100 to about 5.0 percent by weight of water; which composition is an antifoam composition.

The choice of emulsifier is governed by two considerations: 1) miscibility in the silicone, and 2) the HLB ratio. It is necessary that the emulsifiers be miscible with the silicone phase in order to provide a water-in-oil emulsion that inverts upon dilution to an oil-in-water emulsion. Miscibility in the silicone is determined by a simple test wherein the silicone and the emulsifier are mixed and heated to between 30 and 50 °C. The

mixture is held in this temperature range until a homogeneous mixture is obtained. Then the mixture of emulsifier and silicone is cooled to room temperature. Applicants define a silicone miscible emulsifier as that emulsifier which forms a homogeneous mixture with the silicone after heating to between 30 and 50 °C followed by cooling to room temperature. Clearly, an emulsifier that does not form a homogeneous mixture is not miscible. While some emulsifiers will satisfy the criterion of this test independent of the silicone used, other emulsifiers may be more or less satisfactory depending on the viscosity of the silicone. The second condition imposed on the emulsifying agent is that the water-in-oil emulsion initially formed must invert to an oil-in-water emulsion upon dilution with water. Thus miscibility and the ability to invert the emulsion upon dilution provide the necessary and sufficient conditions to define the emulsifier.

A preferred class of emulsifying agents are the ester derivatives of oleic acid. Thus for example oleic acid mono-, di, and tri-glycerides, polyethylene oxide sorbitan mono-, di-, and tri-oleates and the like are preferred emulsifying agents i.e. oleate surfactants.

Within the range of HLB values that are most preferred for the emulsifier, the oleate derivatives having the general formula:



, where each Q is independently defined by the formula $-(\text{CR}^{11}\text{R}^{12}\text{CR}^{13}\text{R}^{14}-\text{O})_n-\text{H}$ where R^{11} , R^{12} , R^{13} , and R^{14} are either hydrogen or one to ten carbon atom monovalent hydrocarbon radicals and n ranges from 0 to about 100, have been found to be particularly effective.

The most preferred range of the HLB ratio is from 5 to 14. As this range is increased at its extremes, a larger number of miscible surfactants

may be employed to prepare a water-in-oil emulsion that inverts to an oil-in-water emulsion upon dilution. However, as the range is widened to values lower than 5 or greater than 14, the stability of the water-in-oil emulsion prior to dilution with water tends to deteriorate.

5 The thickening agents are generally water soluble polymers that may be anionic, non-ionic, or cationic in either solid or liquid form. Generally any appropriate thickening agent may be used as long as the emulsion is not broken by the thickening agent. The thickening agents may be any suitable water soluble polymer that does not break the emulsion. Particularly
10 preferred thickening agents are thickening agents that are water soluble polymers selected from the group consisting of polyacrylates, polyamides, polyamines, styrene sulfonate polymers, polyethylene oxides and cellulose derivatives having a molecular weight ranging from about 100 to about 100,000,00 dalton.

15 The water-in-oil emulsions antifoam agents of the present invention are generally prepared by mixing a silicone, a water-soluble polymer, emulsifiers, water and an optional neutralizing agent. The following examples are provided to illustrate the invention and are not intended in any to limit the scope of the appended claims. All U.S. patents referenced herein
20 are herewith and hereby incorporated by reference.

Experimental

Examples 1 - 3

Table 1 shows the weights of the various components used to prepare an antifoam composition that can be diluted with water.

Table 1: Antifoam Compositions

| <u>Component</u> | <u>Example 1</u> | <u>Example 2</u> | <u>Example 3</u> |
|--------------------------------|------------------|------------------|------------------|
| weight in grams | | | |
| AF9000™ ⁽¹⁾ | 15 | 15 | 15 |
| Arlacel 186™ ⁽²⁾ | 5 | 4 | 6 |
| Polysorbate 80™ ⁽³⁾ | 5.14 | 6 | 4 |
| SF96-350™ ⁽⁴⁾ | 15 | 15 | 15 |
| EMA10™ ⁽⁵⁾ | 0.47 | 0.47 | 0.47 |

Notes to Table 1:

1. AF9000™ is an antifoam composition containing hexamethyldisilazane treated silica and polydimethylsiloxane, manufactured by GE Silicones, Waterford, New York.
- 5 2. Arlacel 186™ is a mixture of fat forming fatty acid mono- and di-glycerides manufactured by ICI Americas of Wilmington, Delaware.
3. Polysorbate 80™ is a polyethylene oxide (20) sorbitan mono-oleate manufactured by ICI Americas of Wilmington, Delaware.
4. SF96-350™ is a polydimethylsiloxane having a viscosity of 350 centipoise at
- 10 25 °C manufactured by GE Silicones of Waterford, New York.
5. EMA10™ is a polymer emulsion containing copolymer of acrylamide and sodium acrylate manufactured by Chemtall of Riceborough, Georgia.

Example 4

A concentrated antifoam composition was prepared as follows: 30 g of Span 80™ (sorbitan mono-oleate from ICI Americas), 70 g Tween 85™ (polyethylene oxide (20) sorbitan tri-oleate from ICI Americas), 180 g SF96-350 and 20 g of a hexamethyldisilazane treated precipitated silica were mixed and homogenized until uniform. 38 g of the mixture thus prepared was blended with 2 g EM533™, a water-in-oil emulsion of about 40 weight

percent acrylamide and sodium acrylate copolymer. The resulting antifoam concentrate was an emulsion that could be easily diluted with water.

Example 5

A pre-mix consisting of 40.2 g of a mixture of 5 - 15 weight percent hexamethyldisilazane treated fumed silica and 85 - 95 weight percent hexamethyldisilazane treated precipitated silica with 361.8 g of a polydimethylsiloxane having a viscosity of 350 centipoise at 25 °C was homogenized until uniform. 68.0 g of Span 80™ and 12.7 g of Tween 85™ were added to the silicone oil-silica mixture and mixed well. This pre-mix was used to prepare additional antifoam concentrates, and is designated in the later examples as pre-mix A.

Examples 6 - 9

Table 2: Antifoam Compositions

| <u>Component</u> | <u>Example 6</u> | <u>Example 7</u> | <u>Example 8</u> | <u>Example 9</u> |
|--------------------------|------------------|------------------|------------------|------------------|
| weight in grams | | | | |
| Pre-Mix A (Example 5) | 15 | 15 | 15 | 15 |
| SF 18-350 ⁽¹⁾ | 16.66 | 16.66 | 16.66 | 16.66 |
| Span 80™ | 3.12 | 2.64 | 2.04 | 1.53 |
| Tween 85™ | 0.61 | 1.21 | 1.81 | 2.62 |
| EM533™ ⁽²⁾ | 0.2 | 0.2 | 0.2 | 0.2 |

Notes to Table 2:

(1) SF 18-350 is a polydimethylsiloxane having a viscosity of 350 centipoise at 25 °C manufactured by GE Silicones of Waterford, New York.

(2) EM533™ is a water-in-oil emulsion of about 40 weight percent acrylamide and sodium acrylate copolymer manufactured by Chemtall of Riceborough, Georgia.

Example 10

200 g of the pre-mix A (example 5) was mixed with 26.41 g of Span 80™ and 23.73 g Tween 85™ followed by the addition of 249.87 g of SF 18-350 to make pre-mix B.

Example 11

To 300 g of pre-mix B was added 6.1 g of Pemulen TR-1™, 2.30 g of 50 weight percent aqueous NaOH and 5 g water. Pemulen TR-1™ is a polyacrylic acid manufactured by B. F. Goodrich of Cleveland, Ohio. The preparation was dilutable with water.

Example 12

To 10 g of Pemulen TR-1™ was added 96 g of water to prepare an aqueous slurry having 9.43 weight percent Pemulen TR-1™. To 22 g of the aqueous slurry of Pemulen TR-1™ was added 200 g of pre-mix B followed by the addition of 0.65 g of 50 weight percent aqueous NaOH. The preparation was dilutable with water.

Example 13

A large reaction kettle was charged with 532.6 pounds of SF 18-350, 62 pounds of a mixture of 5 - 15 weight percent hexamethyldisilazane treated fumed silica and 85 - 95 weight percent hexamethyldisilazane treated precipitated silica and 0.6 pounds of oleic acid. The mixture was heated to 120 °C while mixing. After stirring for four hours at 120 °C the mixture was cooled to 25 °C. 24.8 pounds of Pemulen TR-1™ was subsequently added and the mixture mixed until uniform. The resulting mixture was homogenized through a Gaulin mixer into a second kettle. To the second kettle containing the homogenized mixture was added 202.8 pounds of Span 80™, 107.8

pounds Tween 85™, and 953.2 pounds SF 18-350 followed by mixing for two hours. While the mixture was being mixed, 62 pounds of water at a temperature of 20 - 35 °C was added. After the first hour of mixing 8 pounds of Proxel GXL™, 2 pounds of methylparaben, and 2 pounds of propyl paraben were added as a biocide package. When all of the materials had been mixed in 42.2 pounds of 10 weight percent aqueous NaOH were added.

This preparation was diluted with varying amounts of water as shown in Table 3.

Table 3: Dilution of Antifoam Concentrate from Example 13

| Designation | Parts Water, Parts by weight | Example 13 Preparation, by weight | Total Parts, by weight | Silicone level, weight percent |
|-------------|---------------------------------|---|---------------------------|--------------------------------------|
| 13A | 1 | 15 | 16 | 5 |
| 13B | 1 | 7 | 8 | 10 |
| 13C | 1 | 3 | 4 | 20 |
| 13D | 1 | 1.66 | 2.66 | 30 |

The viscosity of the various diluted preparations of example 13 was adjusted by the addition of 10 weight percent aqueous acetic acid. Sufficient acetic acid solution was added to change the pH which resulted in changing the viscosity of the diluted concentrate. As shown in Table 4, relatively small changes in pH level produce significant changes in the viscosity of the antifoam concentrates.

Table 4 Viscosity pH Relations for Antifoam Concentrate

| <u>13B</u> | | <u>13C</u> | | <u>13D</u> | |
|------------|----------------------------------|------------|----------------------------------|------------|----------------------------------|
| <u>pH</u> | <u>Viscosity,</u> cps at 25°C | <u>pH</u> | <u>Viscosity,</u> cps at 25°C | <u>pH</u> | <u>Viscosity,</u> cps at 25°C |
| 5.80 | 8,870 | 5.40 | 18,700 | 6.15 | 22,600 |
| 5.22 | 4,070 | 5.00 | 9,400 | 5.25 | 14,800 |
| 5.15 | 815 | 4.59 | 2,400 | 4.48 | 11,400 |
| 4.97 | 230 | 4.40 | 675 | 4.30 | 3,700 |
| | | | | 4.10 | 1,900 |
| | | | | 4.03 | 1,150 |

Claims

1. A water-in-oil emulsion comprising:

(a) from about 1 to about 99 percent by weight of a polyorganosiloxane of the general formula



5 where $M = R^1R^2R^3SiO_{1/2}$ where R^1 , R^2 , and R^3 are each independently selected monovalent one to forty carbon atom hydrocarbon radicals

where $D = R^4R^5SiO_{2/2}$ where R^4 and R^5 are each independently selected monovalent one to forty carbon atom hydrocarbon radicals, with the stoichiometric subscript, x , ranging from about 1 to about 10,000 wherein the
10 viscosity of the polyorganosiloxane ranges from about 1 to about 10,000,000 centipoise at 25 °C;

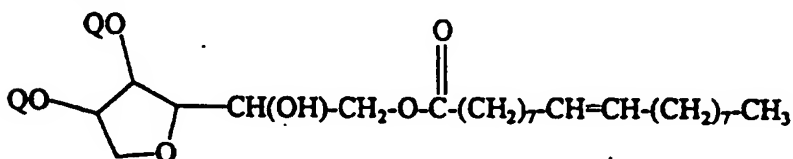
(b) from about 0.01 to about 50.0 percent by weight of a finely divided silica having a size ranging from about 0.001 to about 1,000 microns average particle diameter;

15 (c) from about 0.10 to about 50.0 percent by weight of an emulsifier or mixture of emulsifiers having on average a hydrophilic lipophilic balance ratio ranging from about 2 to about 20; and

(d) from about 0.001 to about 20.0 percent by weight of a thickening agent or mixture of a water soluble thickening agent selected from the group
20 consisting of polyacrylates, polyamides, polyamines, styrene sulfonate polymers, polyethylene oxides and cellulose derivatives having a molecular weight ranging from about 100 to about 100,000,00 dalton; and

(e) from slightly greater than 0 to about 20.0 percent by weight of water; wherein when said water-in-oil emulsion is added to a quantity of
25 water greater in weight than the weight of said water-in-oil emulsion said emulsion inverts to an oil-in-water emulsion wherein said oil-in-water emulsion reduces foaming.

2. The water-in-oil emulsion of claim 1 wherein the emulsifier has a hydrophilic lipophilic balance ratio ranging from about 3 to about 18.
3. The water-in-oil emulsion of claim 2 wherein the emulsifier has a hydrophilic lipophilic balance ratio ranging from about 4 to about 16.
4. The water-in-oil emulsion of claim 3 wherein the emulsifier has a hydrophilic lipophilic balance ratio ranging from about 5 to about 14.
5. The water-in-oil emulsion of claim 4 wherein the emulsifier is an ester of oleic acid.
6. The water-in-oil emulsion of claim 5 wherein the ester of oleic acid has the formula:



, where each Q

is independently defined by the formula $-(CR^{11}R^{12}CR^{13}R^{14}-O-)_n-H$ where R^{11} , R^{12} , R^{13} , and R^{14} are either hydrogen or one to ten carbon atom monovalent hydrocarbon radicals and n ranges from 0 to about 100.

7. The water-in-oil emulsion of claim 5 wherein the ester of oleic acid comprises an ester of glycerin.
8. The water-in-oil emulsion of claim 5 wherein the ester of oleic acid comprises a mono-ester of glycerin.
9. The water-in-oil emulsion of claim 5 wherein the ester of oleic acid comprises a tri-ester of glycerin.
10. A water-in-oil emulsion consisting essentially of:

(a) from about 1 to about 99 percent by weight of a polyorganosiloxane of the general formula



where $M = R^1R^2R^3SiO_{1/2}$ where R^1 , R^2 , and R^3 are each independently selected monovalent one to forty carbon atom hydrocarbon radicals

where $D = R^1R^2SiO_2/2$ where R^1 and R^2 are each independently selected monovalent one to forty carbon atom hydrocarbon radicals, with the stoichiometric subscript, x, ranging from about 1 to about 10,000 wherein the viscosity of the polyorganosiloxane ranges from about 1 to about 10,000,000 centipoise at 25 °C;

(b) from about 0.01 to about 50.0 percent by weight of a finely divided silica having a size ranging from about 0.001 to about 1,000 microns average particle diameter;

(c) from about 0.10 to about 50.0 percent by weight of an emulsifier or mixture of emulsifiers having on average a hydrophilic lipophilic balance ratio ranging from about 2 to about 20; and

(d) from about 0.001 to about 20.0 percent by weight of a thickening agent or mixture of a water soluble thickening agent selected from the group consisting of polyacrylates, polyamides, polyamines, styrene sulfonate polymers, polyethylene oxides and cellulose derivatives having a molecular weight ranging from about 100 to about 100,000,00 dalton; and

(e) from slightly greater than 0 to about 20.0 percent by weight of water; wherein when said water-in-oil emulsion is added to a quantity of water greater in weight than the weight of said water-in-oil emulsion said emulsion inverts to an oil-in-water emulsion wherein said oil-in-water emulsion reduces foaming.

11. A method for reducing foam comprising:

- (a) preparing a water-in-oil emulsion of any preceding claim;
- (b) adding the water-in-oil emulsion to a quantity of water having a weight greater than the quantity of the water-in-oil emulsion; and
- (c) inverting the emulsion from a water-in-oil emulsion to an oil-in-water emulsion whereby the rate of foam collapse exceeds the rate of foam formation.



Application No: GB 9714961.1
Claims searched: 1-11

Examiner: Diane Davies
Date of search: 15 October 1997

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.O): C3T

Int Cl (Ed.6): B01D 19/04; C08L 83/04; C11D 3/00, 3/37; D21H 21/12

Other: Online: CAS-ONLINE, EDOC, JAPIO, WPI

Documents considered to be relevant:

| Category | Identity of document and relevant passage | Relevant to claims |
|----------|--|--------------------|
| A | EP 0658361 A (Th. Goldschmidt AG) Whole document: foam suppressant emulsion comprising polysiloxane, silica and water which may be diluted in water. | |
| A | Abstracts of JP 081922001 A (Sanpuku Co.) Water in oil defoaming composition comprising a polysiloxane, silica, emulsifier, a further organic compound and water. | |

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|---|---|---|--|
| X | Document indicating lack of novelty or inventive step | A | Document indicating technological background and/or state of the art. |
| Y | Document indicating lack of inventive step if combined with one or more other documents of same category. | P | Document published on or after the declared priority date but before the filing date of this invention. |
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